THE CONSERVATION OF SEVENTEENTH CENTURY

ARCHAEOLOGICAL GLASS

A Thesis

by

CORY ARCAK

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF ARTS

August 2009

Major Subject: Anthropology

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Approved by:

Chair of Committee,	C. Wayne Smith
Committee Members,	Cynthia Bouton
	David Carlson
Head of Department,	Donny L. Hamilton

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ABSTRACT

The Conservation of Seventeenth Century Archaeological Glass.

(August 2009)

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Chair of Advisory Committee: Dr. C. Wayne Smith

The primary goal of the conservator is to stabilize and conserve artifacts with the best possible treatment available. Ideally, these treatments are noninvasive and reversible, and maintain the integrity of the object as a top priority. In this respect, it is the responsibility of the conservator to research other possible treatments when traditional methods prove to be insufficient to properly stabilize and conserve an object. Sometimes choosing to treat with a seemingly unorthodox method is the only chance for the objects survival. Though glass is considered one of the most stable archaeological materials, noninvasive, reversible treatments are not always possible given the level of deterioration glass objects undergo within the archaeological setting, specifically the underwater or waterlogged archaeological setting.

This research is a consideration and investigation of the use of silicone polymers and silanes as consolidation materials for 17th-century glass recovered from aqueous environments. Working within the Conservation Research Laboratory and the Archaeological Preservation Research Laboratory at Texas A&M University, a newly developed polymer passivation technique utilizing materials acquired from the Dow

Corning Corporation was applied to archaeological glass recovered from the 1686 shipwreck *La Belle*, excavated in Matagorda Bay off the coast of Texas by the Texas Historical Commission from 1996 to 1997.

The successful application of a hydroxyl ended silicone polymer Q-1 3563, combined with a methyltrimethoxysilane intermediate crosslinker, Q-9 1315, at a 15% solution by weight and catalyzed with dibutyltin diacetate (DBTDA Fascat 4200) occurred in 1999. This project was the first large scale application of silicone polymers and silanes to 17th-century archaeological glass recovered from a marine site. Through this investigation we answered a number of questions regarding the use and application of the silicone technologies and confirmed that these materials are a viable resource for glass consolidation and conservation in terms of the suggested conservation guidelines of the IIC. The silicone technology was successfully applied to numerous types, forms, colors and degradation levels of glass. This included successful application to composite artifacts and the retreatment of objects unsuccessfully treated with a "traditional" method.

Benim Baş Belam

and My Dearest Fred

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To my friends and colleagues at the Conservation Research Laboratory and the Archaeological Preservation Research Laboratory, thank you for the guidance, suggestions, questions and laughter through a time of investigation and experimentation as I learned the ropes and branched out into my own research with silicone polymers. Helen, Jim, John, Taras, Pete, Jon, Mark, Stephanie, Amy, Pete, Ann, Adam and Dan, your support and friendship will not be forgotten.

Thank you to the administration and Friends of the MSC Forsyth Center Galleries. Your support provided an opportunity to share this research with our local and international colleagues and friends in the glass world.

Finally, thank you to all my family and friends in the U.S. and abroad and especially to those that have gone on before. Each of you has played an integral role in the journey of my life. I wouldn’t be where I am without each and every one of you.

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CHAPTER I

INTRODUCTION

With over 5,000 years of production, man-made glass (Tait 1991) has made a profound impact on the cultures of the world, both as an art form and as a functional tool aiding in survival. The conservation of archaeological and historic glass is an integral part of preserving one of the world's nonrenewable cultural resources. Noel Hume (1970) refers to artifacts as "signposts to the past" and "three-dimensional additions to the pages of history." Without proper artifact conservation, the irreplaceable information contained within these recovered "signposts" and "three-dimensional additions" may be lost forever.

With the preservation of archaeological material in mind, the primary goal of the conservator is to stabilize and conserve artifacts with the best possible treatment available. Ideally, these treatments are noninvasive and reversible and the integrity of the object is first priority. In this respect, it is the responsibility of the conservator to research other possible treatments when traditional methods prove to be insufficient to properly stabilize and conserve an object. Sometimes choosing to treat with a seemingly unorthodox method is the only chance for the object's survival.

This thesis follows the style of *Historical Archaeology*.

Though glass is considered one of the most stable archaeological artifacts (Frank 1982, Hamilton 1998, Rodgers 2004), noninvasive, reversible treatments are not always possible given the level of deterioration glass objects undergo within the archaeological setting, specifically the underwater or waterlogged archaeological setting.

In the field of underwater archaeology, the extreme nature of the aqueous environments from which objects are recovered is substantially different from the dry land environments that many archaeologists and traditional conservators are most familiar. During burial, objects are prone to chemical, biological, and physical decay. Primary factors that affect the state of preservation of an object are temperature, burial state, soil composition, ground activity, fauna and flora activity, human activity, the presence of pollutants or microorganisms and moisture levels. For glass, exposure to water is one of the most damaging elements this fragile material can be subjected to, as Frank (1982) notes "of all the usual atmospheric contaminants of glass, liquid water is by far the worst." When exposed to water, an ion exchange process occurs between the surface of the glass and the solution in which it is immersed (Weier 1974, Pearson 1987, Newton and Davison 1989). This ion exchange can result in a very friable material subject to quick dissolution in the aqueous environment. The same can be said once the object is removed from the original burial site. Subjecting the object to a post excavation environment disrupts the state of equilibrium that the object developed during burial (Bowens 2009).

In addition to considering the damage caused by exposure to water, the conservator must also consider the composition of the glass, the structure and form, the manufacturing process, the raw materials used during manufacture, the wear from use, and the environment the object may have been exposed to prior to burial. Any or all of these factors may facilitate or hinder the conservation of the object. Due to the multiple factors listed, challenges to the successful conservation of glass materials are diverse in scope and rise significantly as the factors multiply. Considering the multitude of variables, it is often not possible to successfully conserve a glass object recovered from an aqueous environment using traditional methods. Therefore, it is imperative that material scientists, conservation scientists and conservators continue to pursue alternative methods of conservation that may provide an alternate answer to the difficult question of glass preservation.

The Research

This research is a consideration and investigation of the use of specific silicone polymers and silanes as consolidation materials for 17th-century glass recovered from a marine site. The investigation includes background information on the composition of glass, followed by a brief overview of glass history and an introduction to the artifacts that were treated with the silicone technique. This background information is followed by a chapter on the considerations of conservation and the physical and chemical degradation that occurs in aqueous environments. Before looking specifically at the silicone technique, we review the three most common materials utilized for glass conservation in the past seventy years. These are cellulose nitrate, polyvinyl acetate, and paraloid B-72. Finally, working with the Conservation Research Laboratory and the Archaeological Preservation Research Laboratory, utilizing materials acquired from Dow Corning Corporation, we look at the utilization of silicone polymers and silanes as a conservation technique for the glass recovered from the 1686 French shipwreck *La Belle.*

The stability, aesthetic qualities, visible structural changes, strength imparted to the material, retreatability, effects on various colors of glass, object forms, degradation levels of glass, and the feasibility for application on composite glass artifacts are explored. The research also looks at the pros and cons of utilizing the silicone technique developed at Texas A&M University for archaeological glass based on the recommended treatment guidelines of the International Institute for Conservation of Historic and Artistic Works (IIC) and the published recommendations of glass scientist and glass conservator, Roy Newton and Sandra Davison (1989) respectively. This research serves as a further study into the applicability of silicone polymers combined with silanes as an additional tool in the glass conservator's toolbox and considers whether this newly developed method is sufficiently stable for long-term use.

CHAPTER II

BACKGROUND

Glass Composition

Glass occurs in nature in the form of obsidian, fulgurites and tektites. Obsidian is a product of volcanic eruptions occurring over forty million years ago. Fulgurites are hollow glass tubes formed when lightning strikes a conductive surface such as quartzose sand, silica, or soil. A tektite is a small glass mass formed as a result of meteors crashing into the earth's surface. Much like man-made glass, obsidian, fulgurites and tektites are all masses of silica fused by intense heat, obsidian by volcanic heat, fulgurites by lightning strikes (Edwards 1977, Newton and Davison 1989, Rodgers 2004) and tektites by meteor activity. For thousands of years, man has utilized this naturally occurring material for survival, trade and ornamental purposes.

Glass produced by humans is primarily a combination of three ingredients, silica, an alkali or flux, and a stabilizer. These materials are melted together in a heat resistant container or crucible. Once melted to the consistency of a molten liquid, the material is either cast, poured into a mold or is blown. Glass blowing is accomplished by gathering the molten material onto a pontil or blowpipe and worked into the desired shape through a series of puffs blown into the pipe, followed by handwork, and reheating techniques.

The main ingredient in glass is silica. Silica comes in forms such as sand, crushed quartz, and quartz pebbles and is the most abundant mineral in the earth's crust. Pure silica requires a temperature of over 2300°C (4200°F) in order to melt. This takes a tremendous source of heat, which was not readily available in ancient times. However, when the second ingredient, an alkali, also known as a flux, is added to the silica, the temperature required to melt the ingredients is reduced substantially to approximately 1600°C (2912°F). This reduction in temperature allows artisans to more readily melt the materials and manipulate the molten mass through the various stages of glassmaking. Alkalis commonly used for glassmaking are derived from the ashes of plants and trees and are referred to as soda ash (sodium carbonate) and potash (potassium carbonate).

The third ingredient necessary for glassmaking is a stabilizer. Commonly used stabilizers are calcium oxide and magnesium. This component is invaluable when making glass. It is the stabilizer that helps to bind the materials together and helps produce a more durable material. Without the appropriate level of stabilizer, the glass object will deteriorate relatively quickly when exposed to high humidity or water.

If color is desired, a glassmaker is dependent on the manufacturing process and/or the addition of a fourth ingredient, a metallic oxide. Artisans use a range of oxides in order to produce various colors of glass. That said, it is not unusual to find ancient glass that is colored green or blue, but this color was not produced intentionally. Rather, the color was a result of natural iron or copper impurities in the silica source (Grose 1984, Martin 1989). However, as the technology developed and the glassmakers began to acquire materials with fewer impurities it was necessary to intentionally add pigments in order to produce desired colors. Some common compounds used for color are; iron for green or brown, cobalt for dark blue and copper for light blue and reds, gold chloride for red, and uranium or lead with antimony for yellow.

Throughout history, the main components of man-made glass have remained relatively unchanged. However, various details of the “recipe” and resulting properties and composition of glass have changed numerous times. All individual glass artifacts primarily owe their stability to the proportion and quality of components within the glass composition. The composition and properties of glass are completely dependent upon the country of production, the environmental region of production, the materials and tools available, the desired end product, and the goals and experience of the glassmaker and the glassmaking team (Pearson 1987).

What Is Glass?

Technologically speaking, according to Brill (1962) the scientist’s definition of glass “is a substance in the glassy state, a state in which the molecular units have a disordered arrangement, but sufficient cohesion to produce over-all mechanical rigidity.” Basically, after investigating the molecular structure of glass, scientists have discovered that glass does not fit into any of the “classical states” of matter, such as gas, liquid or solid. Rather, glass is a rigid material with the internal structure similar to a liquid (Frank 1982) Generally speaking, when a liquid becomes a solid the internal molecular structure changes from a random state to an organized molecular structure. This is not the case for glass. As liquid glass begins to cool, its molecular structure remains disordered even as it becomes rigid like a solid. Essentially, glass has a three dimensional network of acidic oxides filled with basic oxides. The basic oxides are not bonded to the network and are comparatively mobile, thus producing the disorganized molecular structure (Pearson 1987).

An Overview of Glass History

The first man made glass is believed to have evolved from the manufacture of faience. Faience is a low-fired mixture consisting primarily of crushed quartz. This mixture has either been previously coated with a colored ceramic alkaline glaze, develops the glaze through efflorescence or forms the glaze when the preformed object is heated (Tite and Bimson 1986, Tite et al, 2007). Archaeological evidence of faience production recovered from Egyptian and Mesopotamian sites dates this material to as early as the fifth millennium B.C. Faience is produced from essentially the same ingredients necessary for the production of glass; silica, alkaline and a base. However, each component is found in varying measures and is subjected to a much lower furnace temperature than what is required when producing glass (Edwards1977, Goldstein 1989).

While technologically glass may have evolved from faience, there have been several legends recorded throughout history as to the actual creation of man-made glass. The following excerpt is taken from Tait (1991), and is one of the most repeated origin tales for the discovery of glass. It is said that Pliny, the Roman elder, recorded the invention of glass as he understood it within his book *Natural History* during the second half of the 1st century A.D.;

> There is a story that once a ship belonging to some traders in nitrum put in here [the coast of modern Lebanon] and that they scattered along the shores to prepare a meal. Since, however, no stones for supporting their cauldrons were forthcoming, they rested them on lumps of nitrum (natural soda) from their cargo. When these became heated and were completely mingled with the sand on the beach a strange liquid flowed in streams; and this, it is said, was the origin of glass.

On the other hand, while Biringuccio, a master craftsman in the practice of smelting and metalworking from the 16th century, retells a similar story that is attributed to Pliny, Biringuccio credits the speculation of “good alchemistic savants” in search of gemstones for the discovery of the material that was neither semi-mineral nor metal, but rather “…a fusible material that is almost made mineral by art and by the power and virtue of fire…” (Biringuccio 1966). And still, another story comes from a volume of 18th-century French works. The volume *Bosc D’Antic*, translated by Cable (2003) attributes glassmaking to lime burners and tile makers. This version of the origin story more closely aligns with the possible evolution process of faience to glass than any of the others, as the tile makers would have been working with faience-like materials in the quest to create the perfect tile.

Despite the unknown origin, according to the archaeological record, artisans were working with man-made glass as early as the Bronze Age, approximately 3000 B.C. in Mesopotamia (modern day Iraq and Syria) and Egypt (Grose 1984), in the form of beads and inlay decorations. Evidence of the first glass vessels dates between 1500 and 1400 B.C. These vessels were created using a technique called core forming. Core formed glass is produced by winding threads of glass around a primarily ceramic center. Once the object is formed, the material is allowed to cool and then the ceramic center is scraped out, leaving a hollow glass object (Edwards 1977, Goldstein 1989). The archaeological record also provides us with evidence of the glass trade circa 1300 B.C., as supported by the Mesopotamian or Egyptian produced glass ingots and beads recovered from the 1300 B.C. Uluburun shipwreck excavated off the coast of modern day Turkey. To date, these glass ingots are the oldest known of their kind (Pulak 1998).

The Bronze Age glass artifacts recovered from sites in Mesopotamia and Egypt are “stylistically indistinguishable…and cannot be unequivocally distinguished on the basis of their chemistry” (Jackson 2005). Thus, it is generally agreed, that either Mesopotamia or Egypt gave rise to the first forms of man-made glass (Goldstein 1989, Cable 2003). The ancient artisans continued advancing the art of glassmaking, experimenting with different recipes, methods, colors, and shapes, all the while producing sought after items such as, amulets, cups, vessels, beads, and bowls for ointments. As glassmaking technology evolved, artisans experimented with and perfected glass-working techniques such as the previously mentioned core forming in addition to, casting, mosaic techniques, molding, pressing and cutting.

Glassmaking became a profitable industry producing high status, luxury items for the wealthy. As Spaer (2001) notes, “a great deal of high-quality glass was produced. At this time, glass was obviously a valuable material, often found in royal or cultic contexts.” Clay cuneiform tablets recovered from the library of King Assurbanipal (668-627 B.C.) in Nineveh, indicate the earliest recording of a glass recipe that may date back to the second millennium B.C. Recorded in the clay is a two-step process that results in a vitreous material that was most likely glass (Oppenheim et al 1988). The artisans of this time were remarkably competent and by all indication, had "mastered complex glass forming and glass coloring techniques" (Spaer 2001). Anyone controlling "the production or consumption [of glass] would have occupied a powerful position." (Jackson 2005).

It was not until the invention of glassblowing and the blowpipe in modern day Syria circa 1 A.D., during the time of the Roman Empire, that the industry was revolutionized. By utilizing the blowpipe, artisans were capable of quickly producing objects as compared to the time consuming method of core-forming. Mass production increased availability and reduced the cost per object. Due in large part to the blowpipe, the availability of luxury, as well as functional glass objects quickly spread throughout Mesopotamia, the Middle East, Africa, and much of Europe, eventually reaching the far corners of the world (Edwards 1977, Battie and Cottle 1997).

***La Belle* and the Recovered Glass**

Prior to the 17th century, glass was primarily a luxury item intended only for the wealthy and affluent. Grose (1984) argues that "glass had been used as a vehicle for elaborate and fantastic works of art, often of very slight practical value and so delicate that their chances of surviving intact for long were extremely limited." As the art form evolved, a compromise was made between beauty and functionality and "the results were the production of aesthetically pleasing relatively robust objects with multiple uses

(Morley-Fletcher 1984). Functional objects such as bottles used to store perishable foods and bottles used for medicinal products became a popular commodity and much more commonplace. What follows describes briefly the glass artifacts recovered from *La Belle.* It is by no means intended to be an in depth analysis of the glass assemblage nor an in depth history of *La Belle.* Analysis and research of the glass assemblage, additional artifacts recovered and the ship, is ongoing and will be compiled by the Texas Historical Commission.

The glass assemblage recovered from *La Belle* is representative of typical 17th century objects used for storing, the transport of goods and trade (Van den Bossche 2001, Bruseth and Turner 2005). The artifacts included, but were not limited to, one intact onion bottle, CRL artifact number 2043, made from a dark green glass also referred to as "black glass" or *verre noir* as seen in figure 1. The original contents of the bottle may have been wine, beer or a pharmaceutical material (Van den Bossche 2001).

Figure 1. Intact onion bottle recovered from *La Belle. Photography courtesy of the Conservation Research Laboratory, Texas A&M University*

In addition, there was one intact apothecary bottle, fragments from other apothecary bottles, fragments from case bottles, wine bottles, and Dutch gin bottles, fragments from case bottles with surviving pewter screw tops and lids and hourglass fragments. For trade with the native cultures of the Americas, the vessel carried pocket mirrors, as seen in figure 2, and over 750,000 glass seed beads.

Figure 2. Fragments of pocket mirrors intended for trade.
Photography courtesy of the Conservation Research Laboratory, Texas A&M University

It is not surprising that these types of glass objects were recovered from *La Belle.* In 1684, *La Belle* set forth on a journey from the French port *La Rochelle*. She accompanied three other vessels, *le Saint François*, *Joly*, and *l'Amiable*. Under the direction of French explorer Rene-Robert Sieur de la Salle, the vessels carried approximately three hundred crew and settlers, and the necessary supplies to sustain life in a new country. Their intended destination was the mouth of the Mississippi river where they were to establish a French colony in the name of King Louis XIV, develop trade with the Native Americans and explore the lower half of the Mississippi (Bruseth and Turner 2005, Roberts 1997).

The expedition was wrought with challenges. Upon reaching the Caribbean after a difficult journey across the Atlantic, they lost *le Saint François* to pirates. Then, as their journey continued, they overshot their destination by over four hundred miles. Finding themselves in what is present day Matagorda Bay off the coast of Texas, the Mississippi river was nowhere in sight. They confronted disease, unfriendly Native

Americans and shortages as supplies began to run short. The colonists and crew became discouraged and lost faith in La Salle and his vision. The *Joly* returned to France with over half of the new colonists and *l'Amiable* ran aground under suspicious circumstances resulting in the loss of numerous supplies (Bruseth and Turner 2005).

In 1686, *La Belle* met with a similar fate. She ran aground during a winter storm taking the remaining supplies with her. Discouraged and distraught, the remaining crew murdered their leader, La Salle (Roberts 1997). The expedition and the installation of a new French colony failed and *La Belle* and her contents seemed lost to the ocean floor forever.

The Texas Historical Commission had an alternative ending to the La Salle saga in mind. In 1996, *La Belle* once again saw the light of day. After years of searching, her final resting place was finally located and excavation ensued. Between 1996 and 1997, *La Belle* underwent a full-scale excavation directed by the Texas Historical Commission. All the objects, including the remains of the ship itself and the cargo (including tools, supplies and the glass assemblage) were excavated and recovered by archaeologists from the Texas Historical Commission who transported them to the Conservation Research Laboratory, Riverside at Texas A&M University in College Station, Texas to undergo stabilization and conservation.

CHAPTER III

CONSIDERATIONS FOR CONSERVATION

Why Conserve?

Conservation is a costly and time-consuming activity. Not all objects recovered from archaeological and historical sites are conserved, just as not every archaeological site is or should be excavated. With limited resources, such as time, money, space and the appropriate conservation technology, many times it is best to leave a site unexcavated or an object insitu until conditions and resources are conducive to a thorough study and proper safeguarding of the irreplaceable materials (Hamilton 1996, Smith 2003). The decision to conserve an object, store an object in an un-treated state, or discard an object is generally agreed upon by the director of the project in cooperation with the conservator (Watkinson and Neal 1998, Keith 2002) and the managers of the eventual repository.

Objects “recovered from a salt water environment are usually well preserved but of a friable nature” (Hamilton 1996) and are particularly vulnerable to loss (Bowens 2009). If the artifacts are not properly conserved in a timely manner they are apt to deteriorate at a very rapid rate” (Hamilton 1996). Without conservation, many artifacts from waterlogged sites would perish, taking with them important historical data. An underlying premise of conservation and archaeology is that the distribution of material culture, as well as its form, physical make-up, and past use have cultural significance indicative of past cultural activities. By studying these remains, we gain considerable insight into our past (Hume 1970, Hamilton1996) and should consider the recovered artifacts as "three dimensional additions to the pages of history." (Hume 1970). Our "past exists only to the extent that the artifacts it created and the landscapes it modified survive into the present" (Keith 2002).

Many times it is only through the archaeological record and the subsequent recovered objects that we can gain knowledge regarding world cultures and economic and social history, specifically knowledge concerning past trades, trade routes, industry, technology, chemistry, craftsmen, art, and fashion. Glass objects that are recovered and investigated, provide an opportunity to learn about 5,000 years of culture and past life-ways that are an integral part of the world's cultural heritage. Through the conservation and maintenance of these objects and by investigating the color, form, chemical make-up, wear patterns, and the location and/or context the object was recovered from, we can continue to elucidate those who came before.

Suggested Guidelines for Choosing a Conservation Method

Logically, we understand that nothing lasts forever and that "conservation science may extend the life of artifacts by decades or even centuries. Developing technology may enable us to preserve entire sites on the seabed, but sooner or later all material objects will yield to corrosion [and/or] decomposition…" (Keith 2002). Therefore, the selection of conservation technologies for stabilizing and conserving material culture is of the utmost importance. As a conservator, applying the conservation material that supports and stabilizes artifacts for the greatest length of time is the most desirable. The durability of glass materials gives the conservator some flexibility when selecting a treatment method. However, this is not always the case (Rodgers 2004).

When choosing a method to conserve archaeological glass, several factors should be taken into account prior to treatment: 1. The type of environment the glass was recovered from: was it a dry land site or a waterlogged site? If it was recovered from a dry land site what was the nature of the burial context? If it was a waterlogged site, was it a saltwater site or a freshwater site? In both instances, what was the object buried near and/or next to? How long was the object buried within the site? 2. The type of glass, as can best be ascertained prior to treatment. 3. The level of degradation the object has already undergone, which cannot be accurately determined until investigated within a laboratory. 4. Any current physical conditions as can best be ascertained prior to treatment. For example, is the glass fractured, cracked, already in a delaminated condition, and/or is iridescence present? What is the stability, body structure, thickness, size of the object and is there surface decoration present? 5. The method of manufacture if known. 6. Any previous treatments applied (if applicable) and 7. What will happen to the glass after treatment? Will it be on display in a museum or gallery? If so, what are the environmental conditions the object will be exposed to? Will it go into storage? If so, again, what are the environmental conditions the object will be exposed to and will the object be handled in the future for research and/or display purposes?

These factors can serve as guidelines for the conservator in deciding what the best approach for treatment should be and what treatment method will be most successful. Though there may be a generalized treatment method proposed, each piece of glass must be analyzed as a separate entity regardless of whether it was recovered from the same burial site or from separate burial sites. No two pieces of glass were manufactured exactly the same way, thus no two pieces of glass should be treated in exactly the same manner.

In addition to the practical guidelines presented above, prior to treating an object, those working in the field of material conservation should consider the ethical guidelines adopted by the International Institute for Conservation of Historic and Artistic Works (IIC). IIC is an independent international organization that "promotes the knowledge, methods and working standards needed to protect and preserve historic and artistic works throughout the world." (International Institute of Conservation 2009). While these guidelines were initially adopted for fine arts conservation, they are equally applicable to the field of archaeological conservation. The following is a selection of these guidelines followed by explanations from Dr. Donny Hamilton, Director of the Conservation Research Laboratory (CRL), Texas A&M University as published in "*Basic Methods of Conserving Underwater Archaeological Material Culture*" (1996):

> 1. Respect for the Integrity of the object - Regardless of an artifact's condition or value, its aesthetic, historic, archaeological, and physical integrity should be preserved. After conservation, an object should retain as many diagnostic attributes as possible.

2. Competence and Facilities - It is the conservator's responsibility to undertake the investigation or treatment of a historic or artistic work only within the limits of his professional competence and facilities.

3. Single Standard - With every historic or artistic work the conservator undertakes, regardless of his opinion of its value or quality, the conservator should adhere to the highest and most exacting standard of treatment. Although circumstances may limit the extent of treatment, the perceived quality or value of the object should never govern the quality of the treatment. All artifacts should receive the same high standard of treatment.

4. Suitability of Treatment - The conservator should not perform or recommend any treatment that is not appropriate to the preservation or best interests of the historic or artistic work. The necessity and quality of the treatment should be more important to the professional then his remuneration. No treatment should be used that is not in the best interest of the object. Any treatment, even though less expensive, extensive, or time consuming, should be avoided if there is a possibility of damaging the artifact.

5. Principles of Reversibility - The conservator should avoid the use of materials that may become so intractable that their future removal would endanger the physical safety of the object. All treatments must be reversible. With this consideration it is understood that no conservation treatment may last indefinitely nor remain superior to all future techniques.

6. Continued Self-Education- It is the responsibility of every conservator to remain abreast of current knowledge in his field and to continue to develop his skills so that he may give the best treatment circumstance permit.

Suggested Guidelines for Glass Conservation

In 1989, leading glass conservators and researchers Roy Newton and Sandra Davison published a work solely focused on the conservation of glass materials. Within this publication the authors compiled an additional list of guidelines conservators should refer to prior to choosing an adhesive, consolidant or other treatment intended to conserve and/or stabilize glass materials. According to Newton and Davison (1989), when choosing an adhesive (consolidant) for glass it is suggested that the material fulfill the following requirements prior to application:

1. Must have a reasonable adhesion to glass so that when they are applied “they will flow and cover the glass so wetting it.”
2. They must “set” to prevent movement of the fragments or the vessels being treated.
3. They must be able to adjust to strains during the set up and after the setting (Shrinkage)
4. It should not put any undue strain on the glass
5. It should be as unobtrusive as possible.
6. It should remain soluble over long periods of time
7. It should be reversible

In the past two decades, much has changed in the world of conservation in regards to the introduction of new technologies, but the ethical guidelines remain relatively unchanged. In the glass world, the most recent suggested guidelines for selecting an adhesive and/or consolidant were published in 2006 (Koob). These guidelines are similar to those listed by Newton and Davison (1989) with the exception of having more specification and including the suggestion that the adhesive and/or consolidant should remain stable for no less than 100 years.

Though the presented guidelines should be referred to and certainly respected, it must also be remembered that they are "guidelines" not mandates and are not practical in every situation. In the field of underwater archaeology, the conservator must be as fluid as the medium the objects are found in and should be as knowledgeable as possible on multiple treatment options. In the case of glass recovered from underwater sites the conservator cannot always ascertain the condition of the glass until after the material is desalinated and dry. Waiting to treat in such cases can result in the loss of the material because many times the internal and chemical damage is not apparent until the object is dry and has started to crumble. At this point it is too late to halt the degradation process.

When choosing a conservation treatment it is necessary to evaluate a material on the permanence of the properties. However, in practice it is necessary to make compromises. Thus, it is wise to consider the IIC guidelines and to choose the most suitable treatment with the least number of disadvantages with regard to the conservation task at hand (Davison and Newton 1989). "Although many techniques have been developed for cleaning, consolidation and preservation, no technique has universal applicability and most should be left to experts with long experience in dealing with a wide range of conservation problems" (Frank 1982). From this platform, we are able to reach farther into the available technologies in an effort to both respect the integrity of the object and to show that we are willing to go beyond the traditional path in an effort to stabilize irreplaceable cultural heritage.

CHAPTER IV

THE DETERIORATION OF GLASS

Environmental Factors

Artifacts are recovered from a variety of environments in the field of archaeology. Within the burial setting objects are prone to chemical, biological, and physical decay. Primary environmental factors that affect the state of preservation of a glass object within the site are temperature, burial state, soil components, ground activity, fauna and flora activity, human activity, moisture levels, (Bowens 2009). Additional environmental factors affecting glass durability and the rate of deterioration are length of exposure, continuous or cycle of attack, the presence of pollutants or microorganisms and marine organisms" (Romich 1999) "marine specimens…present extra problems because of the special environment in which they are found, often at some depth and buried in sediment of complex composition" (Frank 1982). The pH of solutions in contact with the glass and external physical stress also contribute to glass deterioration (Sirois 1999, Rodgers 2004). In particular the aggressive nature of a shipwreck itself contributes to the deterioration of the artifacts prior to burial. *La Belle* was grounded during a storm and likely tossed by the rough seas of Matagorda Bay. Once the ship violently came to rest on the Bay floor partially burying itself under the sediment years would pass as a continual burial took place.

For glass, “ the fact remains with most of these materials [referring to glass, ceramic, and stone artifacts] that entry into the archaeological record, through breakage, disaster, or loss remains the most destructive event visited upon them” (Rodgers 2004). While this has some bearing for dry land sites, for a wet site the dynamics are substantially different. Artifacts recovered from a marine environment are generally well preserved, but tend to be of a friable nature. In a marine environment, glass as any material, reacts with its surroundings. Of all the potential atmospheric contaminants of glass, water is by far the worst (Frank 1982, Newton and Seddon 1999). Water, including water vapor, causes surface deterioration of glass artifacts, particularly those with compositions susceptible to decay (Frank 1982, Goffer 1983, Newton and Davison 1989). Without the presence of liquid water or another form of moisture, stable glass can remain in excellent condition for a number of years. Glass is considered one of the most durable man-made materials recovered from historic sites (Frank 1982) as can be attested to by several examples of centuries old glass recovered from land sites that are now housed in museums around the world, such as the Metropolitan Museum of Art in New York City, New York and the Victoria and Albert Decorative Arts Museum in London, England. Both institutions house substantial glass collections from dry land archaeological sites and a majority of both collections are stable.

Glass Factors

There are additional factors related to the composition and structure of glass that influence the type and rate of glass deterioration. These include “the proportion and quality of raw materials used in manufacturing, the manufacturing technology, the wear from use and the environment (prior to burial) in which the glass was kept, the original structure of the glass, and the composition of the glass. In addition, “the resistance of glass against chemical attack does not only depend on the bulk composition, but also on its thermal history, its homogeneity, the roughness of it surface and any prior surface treatment leading to changes in the surface structure” (Romich 1999). It also depends on the nature of the surface of the glass and the surface area that is exposed to the surrounding environment (Frank 1982). All of these factors affect the durability of glass artifacts in an archaeological setting and should be taken into consideration when discussing the deterioration of glass. As Weir (1974) points out in the article “The Deterioration of Inorganic Materials Under the Sea,” and Pearson (1987) continues, “the exact mechanism” involved in the deterioration of glass in underwater environments is still not fully understood.

When looking at the deterioration of glass within a marine environment it is necessary to understand the phenomenon of diagenesis. Diagenesis is the sum total of physical, chemical and biological changes which sediment undergoes after it is deposited. Colin Pearson (1987) notes that the controlling factors in marine sediment diagenesis are the solid-liquid exchange phenomena, pH, Eh, and organic metabolic processes. These complex interactions are the cause of an ordered sequence of different chemical environments and it is these chemical environments that aid in the decomposition of objects on the ocean floor. According to Newton and Davidson (1989) there is an inward diffusion of water molecules that reacts with oxygen atoms producing hydroxyl ions that migrate out with the alkali cations (causing alkali extraction). During the alkali cation leaching protons replace them to maintain electrical neutrality. The protons are smaller than the cations thus resulting in a surface layer with a smaller volume. The decrease in volume can lead to micro porosity of the surface layer which may cause the multilayered effect found in surface crusts (Newton and Davison 1989).

In the case of glass, a seemingly impervious material, changes occur at the molecular level with an exchange of ions. In submerged glass, sodium and potassium ions move to the surface of the glass. At the surface of the glass, sodium and potassium hydroxides are formed as a result of the interaction of the ions with the salt water. At this point hydrogen ions enter the structure of the glass cutting the bonds within the silica network and a gel layer is formed. The formation of this gel layer results in "thin hydrated layers of glass and physically separated lamellae of amorphous siliceous layers" (Pearson 1987). As this process continues and the hydroxides are exchanged with hydroxides, new potassium and sodium ions leach to the surface continuing the process. This ion leaching and bond breaking degrades the glass leaving an opaque, laminated and weathered surface (Goffer 1983, Pearson 1987, Rodgers 2004) and will continue until the object reaches equilibrium with its surroundings (Minten 1999).

When these objects are buried under sediment as in the case of the glass objects from *La Belle*, “thick hydrated silica layers are formed which lead to the typical iridescent films” (Pearson 1987) commonly observed in recovered glass objects. In principle the gel-layer can act as a protective barrier against further corrosive attack on the bulk glass unless the layer is disturbed by marine organisms, excavation or post excavation activity (Romich 1999).

Glass is considered to be one of the most stable archaeological materials; however it can undergo complex disintegration within the archaeological environment. Evidence of weathering may present itself in the forms of “…dulling, fine cracking, frosting, iridescence, crusting and pitting” (Frank 1982). An example of this weathering can be seen in Figure 3. Many times this weathering is not visible to the naked eye and only becomes apparent once the object is dried.

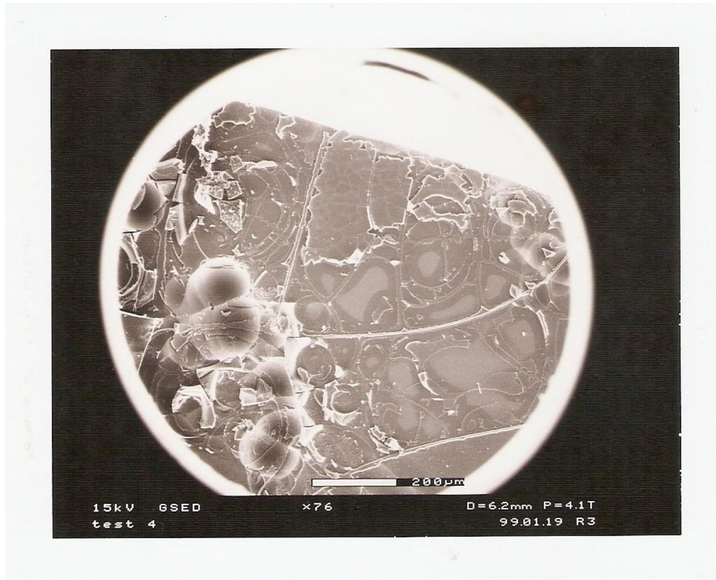

Figure 3. Magnified image of glass recovered from a marine site.
Note the cracking, flaking, and devitrification.

The glass objects recovered from *La Belle* were in various stages of degradation. As in many marine sites, the glass objects were subject to chemical deterioration (as described above), physical deterioration from wave and sediment action and burrowing marine organisms. In one particular example, the marine organisms attached themselves to the surface of the vessel and burrowed under the layers of hydrated glass causing some areas to flake off as seen in Figure 4.

Figure 4. Onion bottle with clamshells prior to silicone treatment. *Photography courtesy of Conservation Research Laboratory, Texas A&M University*

With surface damage caused by the marine organism further chemical deterioration is possible resulting in a less stable object. As the stability of the object is compromised in situ, the likelihood of the object to survive excavation and subsequent conservation decreases. Additionally, further physical damage is possible during survey, excavation, and post excavation. Once an object has been removed from its burial site it can undergo rapid changes in appearance. This is particularly important when removing an object from a waterlogged site. Upon exposure to the sun and air a rapid loss of moisture can occur resulting in the loss of the chemical equilibrium that was established during burial (Frank 1982). Rapid changes in temperature and humidity may cause shrinkage of the gel-layer that formed during burial leading to a micro-porous structure. The increased surface area can continue to react with water and lose contact with the remaining bulk glass. As a result, flaking off of the gel-layer can occur (Romich1999).

Excavation Factors

The Matagorda Bay system is the second largest estuary on the Texas Gulf Coast. It has a high quantity of suspended materials being delivered by the freshwater inflow of the Colorado River, which results in very low visibility. Due to this low visibility and the rough nature of the gulf coast waters the THC decided to excavate *La Belle* behind the safety of a cofferdam. Building the cofferdam around the shipwreck and pumping out the excess water from the center of the cofferdam essentially created a semi-dry land site out of a wet land site. Once the bulk of the water was removed, the excavation team began to implement dry land techniques to remove the archaeological materials. Though they quickly implemented safety measures to protect the fragile site, inevitably, some of the glass objects broke under the stress of the excavation itself.

Another factor contributing to the degradation of artifacts during the excavation was that this once wet site was considerably drier. Exposure to the open air, the sun, fluctuating temperatures and human activity compromised the equilibrium the artifacts had maintained for 300 years and quickened the natural deterioration process. The excavation team had to continuously run water over the site in order to protect the artifacts.

Post Excavation

Another factor that impacts the rate of deterioration is the post excavation environment. Once recovery from an archaeological site occurs it is the responsibility of the archaeologist to ensure the stabilization of the materials. The decomposition process begins almost immediately and must be mitigated (Rodgers 2004). It is at this point that the objects are exposed to a new set of elements furthering the degradation process (Watkinson and Neal 1998). A post excavation environment is generally, rich in oxygen, is generally warmer than the burial site and has a wider range of temperature fluctuations which result in rapid deterioration. The post excavation environment also has higher light levels exposing the artifacts to damaging ultra violet light, and is rich in bacteria, fungal spores, and chemical and biochemical contaminants.

Probably one of the most damaging post excavation factors is the improper handling of the artifacts by untrained individuals. Though glass is generally durable, post excavation, this durability may only be one of appearance. Conservators do not know what level of decomposition has occurred with a glass object until they are able to fully analyze the piece in a laboratory. Each time a glass piece is handled prior to conservation the likelihood for internal damage increases.

CHAPTER V

OVERVIEW OF TRADITIONAL METHODS

Throughout the field of conservation, there have been a number of materials used as an adhesive and/or consolidant in an effort to stabilize archaeological glass. An adhesive bonds two surfaces together while a “consolidant is designed to harden inside a porous material making it more robust” (Cronyn 1990). Consolidation is a widely used method for the conservation of deteriorating artifacts manufactured from various materials. The immediate concern is the breakdown and/or deterioration of the internal structure of the object. The method selected must be capable of impregnating the object in order to import strength by binding with the structure (Romich et al 1995). It is understood within the field of conservation that “only those delicate and friable artifacts that are actually losing surface detail should be considered for consolidants” (Rodgers 2004) and that consolidation is based on the idea that the object is deteriorating at a rate that if left untreated, the object will not survive (Charola et al 1986). The following are brief summaries of the three most commonly used materials for glass conservation in the 20th century; cellulose nitrate, polyvinyl acetate (PVA) and Paraloid B-72.

Cellulose Nitrate

Cellulose nitrate or nitrocellulose, was discovered in 1833. It has been used in conservation since the late 19th century and was the adhesive of choice for archaeologists from approximately1930 to 1970 (Shashoua et al 1992, Neiro 2003). Cellulose nitrate is produced commercially and can be found at the local hardware store under the name *Duco Cement*. When conservation materials are produced commercially the conservator must use caution before implementing treatment. As a commercial material, the contents will vary as to the availability of raw ingredients and the needs of the manufacturing entity. This leaves the conservator always questioning the actual components of the adhesive and whether these components are harmful to the artifacts.

Cellulose nitrate was and still is used in laboratories for the repair of glass and ceramic objects. At the time of most prevalent use, cellulose nitrate was seen as an answer to conservators questions of quickly and painlessly mending glass and ceramic objects. It was relatively inexpensive, bonded well with glass and ceramic surfaces, was easy to use, was strong and was clear, thus produced “seamless” repairs (Shashoua et al 1992).

Today it has been found to be unstable as an adhesive, degrades spontaneously, produces gaseous nitrogen oxides which form corrosive nitric acid in the presence of moisture, is light and heat sensitive, is brittle and yellows as it ages (Moyer 1982, Selwitz 1988). When used with glass materials, the glass materials “transmit light to the polymer, and light transmission has long been known to provide an additional mechanism of decomposition” (Selwitz 1988). Despite these findings, some conservators continue to use the material in the field and laboratory (Jamestown interview 1999, pers.comm., Neiro 2003).

Polyvinyl Acetate

Polyvinyl acetate (PVA) is used both as an adhesive and as a consolidant for archaeological glass and ceramic materials. It is a clear emulsion and/or resin that can be prepared in the laboratory to the consistency desired by the conservator. PVA is inexpensive, easy to work with and readily bonds with the glass surface. It has been used as an alternate to cellulose nitrate since the mid 1970's (Newton and Davison 1989) and continues to be a relatively common conservation resource today.

Through laboratory investigations this material has been found to "release appreciable amounts of acetic acid," (Down et al 1996) and loses its strength and flexibility over time. In addition, it may become "tacky" at high temperatures or in the most severe cases, the joins will sag and/or the adhesive will run out of the join (Cronyn 1990). As the material ages, it is susceptible to distortion and movement also known as cold flow. This is combined with a tendency to attract dirt, shrink and turn yellow after a long period of time.

Paraloid B-72

Paraloid B-72 is a thermoplastic acrylic resin used as both an adhesive and a consolidant for glass and ceramic materials. In the early 1980's paraloid B-72, also referred to as Acryloid B-72 in the United States, was suggested as an alternative to PVA for use in the conservation of friable materials due to its stability to light and heat-aging (Koob 1986). Currently, paraloid B-72 is the most commonly used conservation material for archaeological glass recovered from dry, as well as underwater sites (Koob 1986, Pearson 1987, Helen DeWolf 1998, pers. comm., Hamilton 1998, C. Wayne Smith 1998, pers.comm., Steve Koob 1999, pers. comm., Lisa Pilosi1999, pers. comm., Chapman and Mason 2003, Paterakis 2003) and is the "standard glue" used in the Conservation Research Laboratory, Texas A&M University (Hamilton 1998).

Paraloid, like PVA, is relatively inexpensive, easy to work with, is clear, produces consistent results and can be prepared in the laboratory to the desired consistency of the conservator. In addition, fragile objects can be stabilized with paraloid B-72 during the desalination process (Hamilton 1998).

Though it has had a long history of positive results, in some cases, paraloid B-72 has proved unstable when used as an adhesive with large ceramics (Shashoua et al 1992). As a consolidant, Paraloid has not always been successful in the consolidation of the most fragile of archaeological glasses recovered from waterlogged sites, tends to be glossy if not applied appropriately and sometimes changes the color of the objects that receive application (Romich et al 1995). Some supporters of Paraloid B-72 suggest that these failures are isolated and likely due to the inexperience of the conservator rather than an inefficiency in the material (Steve Koob 2007, pers. comm.).

Discussion

As noted by Feller (1967), when a material satisfies enough requirements it is widely recommended for use until another material supersedes it in performance. This seems to be the case for each of the three materials mentioned in this chapter. When each material was first introduced to the conservation field it appeared to fulfill most, if not all, of the guidelines for selecting a conservation material as listed by Newton and Davison: they reasonably adhered to glass, set to prevent movement, appeared to adjust to strains, did not put undue stress on the glass, were unobtrusive, remained soluble for a long period, and in remaining soluble, were considered “reversible.” And each material appeared to be an improvement from the past material. However, as time has passed and each material has been tested by environmental constraints and in the laboratory, concerns of long-term stability have developed. Cellulose nitrate has all but fallen out of favor as an adhesive and has never been successfully applied as a consolidant. PVA, while still used, is not necessarily the best choice of consolidant for glass objects because it may yellow as it ages and it attracts dirt, thus we now have paraloid B-72. However, even paraloid B-72 has its challenges.

We are creatures of our environments and we do tend to utilize the materials that make us the most comfortable until someone can convince us there is something better or that there may be an alternative. As has been indicated in the brief summaries, despite their challenges, these three materials continue to have their purpose and place in the conservation world. However, these materials do not solve all of our problems nor are applicable to every situation. It is imperative to continue searching for new solutions to the age-old problems conservators are confronted with when treating archaeological glass.

CHAPTER VI

SILICONE CONSERVATION METHOD

The silicone method is a passivation polymer process with a crosslinker used as a consolidation method to conserve and stabilize archaeological materials removed from waterlogged sites. To date, the method has primarily been utilized for organic remains, though application has been investigated on a wide array of materials such as glass and metals (Smith 1998, 2003).

History of the Method at Texas A&M University

In regards to silicones and silanes, both materials have been investigated and or referred to as individual materials for conservation ranging from organic objects to stone (Charola et al 1986, Dinsmore 1987, Horie 1990, Coghlan 1997, Miller 2001, Smith 1997) and in some cases specifically for glass (Errett et al 1984, Newton and Davison 1989, Romich et al 1995, France Remillard 1999, pers.comm., Davison 2003, Smith 2003, Koob 2006) but, with the exception of ongoing research in the APRL and CRL by Dr's Wayne Smith and Helen DeWolf and conservation graduate students at Texas A&M University, very little research has been conducted or is published citing the use of silicone polymers combined with silanes as a glass conservation method.

This research began in 1997 when Dr. C. Wayne Smith of the Archaeological Preservation Research Laboratory in conjunction with Dr. Donny Hamilton of the Conservation Research Laboratory in the Nautical Archaeology Program at Texas A&M University began investigating alternative methods for the conservation of artifacts from marine sites. The investigation and application of this method have resulted in two U.S. patents for silicone polymer and silane treatments of organic and inorganic waterlogged archaeological materials. Initial experiments with silicones and silanes as a glass conservation material were conducted on 17th- century English onion bottles recovered from the sunken city of Port Royal, Jamaica. The results were promising and have led to further development and testing of the technique, including initiating the research in this thesis.

Treatment of *La Belle's* Glass

Working within the Conservation Research Laboratory and the Archaeological Preservation Research Laboratory, a newly developed silicone technique utilizing materials acquired from Dow Corning Corporation, were applied to most of the glass assemblage recovered from *La Belle*[1]. Because we were unsure of the level of degradation and devitrification the objects had undergone during burial, rather than risk unnecessarily losing any of the objects, it was decided to consolidate all the glass materials in the assemblage. Prior to the application of the silicone technique the appropriate artifact documentation, desalination and dehydration processes for the material were completed.

Arrival to Texas A&M

Conservation begins from the moment the object is removed from the archaeological site and frequently before it is removed. A preliminary assessment of the

[1] The over 750,000 glass trade beads were not included in this study.

condition of the object was made while the glass artifacts remained insitu. Following assessment the most beneficial and least destructive method of extraction was determined prior to removal. This phase of the process is especially important for the most fragile of objects such as glass.

After each artifact was assessed and recorded insitu for future analysis, the archaeologists carefully removed each object from its 300 year old resting place within the wreck. Upon removal, the glass was assigned a unique artifact number, gently rinsed if necessary, and placed into a container of salt water with the appropriate packing materials. In the case of objects recovered from an underwater and/or waterlogged site it is extremely important to keep the object wet. If the object were to begin to dry before the chlorides were removed it is possible that essential diagnostic features may be lost and the objects integrity would be compromised (Pearson 1987, Bowens 2009). A second reason for keeping the object wet is to maintain structural integrity. Though a preliminary examination is undertaken insitu, it is not possible to firmly verify the condition of glass objects recovered from wet sites (Minten 1999). These containers were then shipped to the Conservation Research Laboratory at Riverside Campus Texas A&M University, College Station Texas for further documentation, cleaning, and conservation.

Desalination

Upon arrival to the lab, the objects were repackaged into sturdier materials and stored in tanks of water in order to begin the desalination process. Each piece of glass was sequentially washed in bathes of water decreasing the salt content. The objects were transferred from salt water to increasingly fresh water in order to remove all the chlorides from the materials and to allow for proper conservation. All glass pieces must be thoroughly washed to remove traces of chemicals and contaminants, in particular chlorides (Pearson 1987, Rodgers 2004) Any salts present, may crystallize and cause disruption of the glass layers (Pearson 1987) During this process the objects were under observation and were monitored with Mercuric Nitrate titrations to assess the chloride levels while the baths were slowly changed from 100% saltwater to 75% saltwater 25% tap, 50% saltwater 50% tap water, 25% saltwater 75% tap water to 100% tap water. The tap water baths were followed by a succession of rainwater baths in a similar sequence,75% tap water 25% rainwater continuously following this system until the object ended up in 100% deionized water. The objects remained in 100% deionized water until the mercuric nitrate indicated a chloride level of 5ppm. During desalination, the most unique pieces of glass were photographed. All the objects were recorded on artifact cards recording size, dimensions, condition, object type if apparent, and were accompanied by an artifact drawing.

Dehydration Process

Once all the chlorides were removed, the glass was dehydrated in successive baths of solvents following the standard dehydration system used at the Texas A&M University Conservation Research Laboratory. This process was done slowly in sequential bathes of varying percentages of solvent and water. The initial bath was 25% ethanol 75% deionized water, followed by 50% ethanol 50% deionized water, then 75% ethanol 25% deionized water to 100% ethanol. The ethanol solvent was followed by a succession of acetone baths as follows, 75% ethanol 25% acetone, 50% ethanol 50% acetone, 25% ethanol 75% acetone to 100% acetone. All glass artifacts were dehydrated with the same process prior to consolidation.

Test Samples

Prior to full scale application of this process three different centistoke silicone polymers were tested in solution to determine which viscosity was most beneficial for glass artifacts. I needed to determine three things, 1. Whether the silicone technique would work with these particular chemicals 2. Whether these particular chemicals would strengthen the glass and 3. Determine which polymer length was the most aesthetically appropriate for glass materials. Prior to this experiment, the selected crosslinker had not been applied to glass and only one of the silicone polymers (SFD-1) had been tested with archaeological glass. The chemicals investigated in this experiment are listed in Table 1.

Table 1. Chemicals used for glass conservation tests and application

Silicone Polymer	Q-1 3563
	SFD-1
	SFD-5
Crosslinker	Q-9 1315 (MEOH/MTM Intermediate)
Catalyst	Fascat 4200- dibutyltin diacetate (DBTDA)

The three polymers tested were Q-1 3563 a short centistokes polymer with low viscosity, SFD 1 a medium centistoke, medium viscosity polymer, and SFD 5 a long centistokes polymer that is extremely viscous. In the CRL under a fume hood, each polymer was individually combined with 15% crosslinker Q-9 1315 by weight in separate disposable cups. One small sample of 17th-century archaeological glass was placed into each solution. Each sample in solution was then placed in a vacuum chamber for fifteen minutes at 26psi. Following the vacuum, each sample was allowed to rest in solution for ten minutes and was then gently removed from the solutions and drained on lint free paper towels. After the bulk of the solution was drained, the glass samples were cleaned with lint free towels and cotton swabs to remove any remaining polymer. The samples were then placed into individual zip lock bags with a wadded paper towel that had been dipped in FASCAT 4200 (DBTDA) in each. In this manner the silicone solution was allowed to catalyze through a vapor method. The samples were left overnight in the catalyst vapor and observed the next morning to determine which polymer length was most beneficial for glass conservation.

Upon visual observation, it was determined that all three polymer chains were successful in consolidating the glass samples and were successful in strengthening the matrix of the glass samples. As far as aesthetic properties, SFD-5 resulted in a dull finish and left a rubbery film on the surface of the glass and appeared to over impregnate the fragile layers of the devitrified glass sample. I would only suggest this polymer if the goal was to completely encase the object, protecting it within its skin of silicone. SFD-1 enhanced the surface color and left a light rubbery film on the surface, resulting in a satisfactory yet average appearance. I would suggest this polymer for general use and to bulk more fragile materials that were going to be handled frequently. I would also suggest SFD-1 if Q-1 3563 was not available. Q-1 3563 was the most aesthetically appropriate polymer for use with this glass. This polymer enhanced the surface color and did not leave a rubbery film behind. The result was satisfactory and aesthetically pleasing. With successful application, it would be difficult for the unaware observer to know that a consolidant had been applied to the glass. I would suggest this polymer for use with all glass artifacts.

Intact Onion Bottle

The onion bottle (CRL artifact #2043) was the only large intact bottle recovered from the site. As mentioned previously, due to the nature of glass degradation in water environments we were not sure of the internal condition of the vessel. In order to be sure the vessel was properly stabilized we decided to consolidate with Q-1 3563. This process was somewhat different than the experiments conducted to test the various centistokes polymer chains. With this object I followed the guidelines as presented by Dr. Wayne Smith (1998). Following standard desalination and dehydration, the vessel was placed in a solution of Q-1 3563 with 15% Q-9 crosslinker by weight. The submerged vessel was placed under a vacuum of 26psi for five hours on day one, followed by eight hours on day two. The vessel was removed from the vacuum chamber and drained over an empty container until most of the excess polymer was removed. After draining the excess solution, catalyst was applied topically and swished inside the vessel to ensure full coverage. The vessel was then placed into a large ziploc bag with vapor catalyst. The bag with the vessel and the catalyst was then placed inside of a warming oven and left overnight as was suggested by Smith (1998). For the next two days the vessel was removed from the Ziploc bag and dipped into the silicone solution, drained and put back into the bag with the catalyst vapor and returned to the oven. After the third day, the vessel was fully consolidated and safe for handling.

Basic Guidelines for the Silicone Process Using Q-1 3563 and Q-9 1315

After successfully determining the most appropriate silicone polymer for glass and successfully consolidating artifact number 2043, the newly developed silicone technique was applied to the remaining objects from *La Belle's* glass assemblage. There were two changes made to the process; 1. I did not use the warming oven and 2. I used solutions of 10%, 12%, and 15% by weight in an effort to test the parameters of the crosslinking solution. Before beginning the process it was necessary to gather the supplies and access the appropriate equipment as listed in Table 2.

Table 2. Supplies for silicone process

Equipment	Well ventilated room with a fume hood
	Vacuum chamber
	Scale for weighing chemicals
	Time keeper
Supplies	Safety goggles
	Disposable gloves
	Disposable cups or other appropriate container to fit the object
	Paper towels or newspaper
	Tweezers and other dental tools for handling and cleaning glass
	Tongue depressors for mixing
	Lint free cloth
	Cotton swabs
	Ziploc bags
Chemicals	Q-1 3563
	Q-9 1315
	DBTDA Fascat 4200

Many of the materials used during this process may be disposable for easy clean up. Mix the selected silicone polymer with 12% to 15% Q-9 crosslinker by weight in a disposable cup. Use a tongue depressor to thoroughly mix the solution. Once mixed, remove the artifact from the dewatering bath and carefully immerse the object into the silicone solution. Once the object is immersed and the initial bubbling has ceased, place the cup with the object into the vacuum chamber. Vacuum the object in the cup between 20 and 26psi depending on the condition of the glass. Vacuum the object until all bubbles have ceased. The cessation of bubbles will indicate that the acetone has been replaced by the silicone oil/crosslinker solution and that the solution has entered within the matrix of the object coating all exposed surfaces and filling layers, holes and microscopic damage. You will see an immediate change in the appearance of the object when placed into the silicone. This is due to the refractive nature of aqueous solutions. Continuous observation is necessary during this step in order to prevent any unnecessary disturbances within the glass. If excessive bubbling occurs reduce the vacuum pressure immediately. This will allow for a more gentle replacement of the acetone helping to prevent further damage and unnecessary flaking to the object. Once the bubbles have ceased allow the piece to remain under vacuum for 30 minutes. This will ensure complete coverage of the object by the silicone. Once vacuum is complete remove the cup with the solution and object from the chamber and allow it to rest at ambient pressure for 10 minutes. Once rested, use the tweezers to carefully remove the object from the solution. Drain the object on paper towels. After thoroughly draining the object wipe clean with cotton swabs or lint free cloth to remove excess polymer. Then place the object on a paper towel and into a Ziploc bag with a wadded paper towel that has been dipped in catalyst. Leave the object in the vapor bag overnight. The next day, replenish the catalyst and leave overnight once more. Be sure to write down any observations and treat each object as an individual piece.

Due to the nature of the glass composition, each object will respond differently to the treatment. Conservatively, the entire process takes approximately two days depending upon the object itself. It should be remembered that these are only basic guidelines for the silicone technique. This is a flexible material and the guidelines should be altered as is necessary for the laboratory utilizing the materials and as is necessary for the objects undergoing treatment.

CHAPTER VII

FINAL DISCUSSION

Overall Results

The silicone technique was applied to 133 artifact numbers with a total of 511 pieces of archaeological glass (see Appendix A). The glass assemblage varied in size, color, form and was representative of degradation levels ranging from poor to good. Overall, there were no adverse effects to the glass assemblage after the application of the materials. The glass was structurally sound and stability was enhanced with the application. The silicone imparted strength to the glass, allowing for handling. The silicone is clear and was applied in such a manner that the treatment is not obvious to the untrained eye.

10%, 12% and 15% Solution

The crosslinker was added to the silicone polymer beginning with 15% by weight. When using the MTM crosslinker for the silicone technique, the standard solution is 3% by weight of the chosen silicone polymer. Q-9 1315 (MEOH) is diluted with alcohol approximately five times more than MTM, thus in order to create a similar crosslinking affect 3% was multiplied by five to reach the average of 15%. A 15% solution does work and did work with all artifacts it was treated with. However, in order to test the parameters of the Q-9 crosslinker, I tested a 10% solution and a 12% solution by weight. The 10% solution does not appear to be enough crosslinker for all glass objects. It does work with some objects that are in the good range of condition, but does not seem to work with all objects that are in the fair to poor range of conditions. Ten artifact numbers did not respond to the 10% solution and exhibited signs of further degradation once exposed to the open air (see Appendix A). These ten artifacts required a retreatment with a 15% dip bath after the original standard procedure in a 10% solution. Thereafter, I tested the 12% solution. All objects responded well to the 12% solution without issue. This testing allowed me to stretch the parameters of the crosslinker and to discover that retreatment with a silicone solution was possible.

Removal of Concretion/Shells

Some glass objects had concretions and clamshells adhered to their surface (figure 4). During the desalination phase, the surface of the glass objects were cleaned as adequately as possible without damaging the glass. The strength of some of the clam shells were such that complete removal prior to consolidation would have damaged the surface. In the case of the onion bottle, I removed as much exterior material as possible prior to dehydrating and consolidation. Once the object was fully consolidated I discovered that the silicone polymers had softened the calcareous material to such an extent that I was able to further remove the shells without harming the glass. The more difficult areas were cleaned with a coarse hair brush and a dremel tool. In some areas calcareous material had imbedded itself within pits in the glass surface. These areas were left untouched as digging the material out of the pits would have compromised the object as a whole. In addition, it was determined that the calcareous material did not impede the consolidation process. After the areas were cleaned I was able to confirm that the glass surface below the clamshell was consolidated.

Discovering that the polymers softened the calcareous materials proved to be an unanticipated asset. With this knowledge, I was able to postpone cleaning until after the objects were consolidated. After consolidation, the objects were more stable and easier to handle as compared to their condition prior to treatment.

Retreatability Example

In the case of the hourglass shown in figures 5 and 6, the head conservator attempted the use of paraloid B-72 as a treatment method. The hourglass was in poor condition and was not expected to survive conservation. It appeared that the water and surrounding sediment were the only things holding the glass together. Upon application, the glass did not respond to the treatment with paraloid B-72. It remained extremely fragile and in an unconserved state. The hourglass was placed back into the alcohol solution to await further treatment.

Having previously been desalinated and dehydrated the next step was to attempt partial cleaning and consolidation once again. The object was in such a fragile state that the edges were gelatinous and the walls of the glass were no longer firm. The walls of the hourglass waved with the movement of the solution when disturbed. Extreme care was taken to apply the silicone method with the understanding that this one attempt may be the only attempt possible to conserve this object. Excess sediment was carefully removed with dental picks, brushes, and gentle movements with the hands. Once cleaned to the necessary level, the object was placed in the silicone solution and underwent the basic silicone treatment. During the draining and catalyst application, a thin layer of silicone was left inside the hourglass to provide additional support. There were no difficulties applying the silicone treatment after the object had received the paraloid B-72 treatment. The completed hourglass is shown in figures 5 and 6.

Figure 5. Hourglass after treatment
Photography courtesy of the Conservation Research Laboratory, Texas A&M University

Figure 6. Opposite side of hourglass after treatment
Photography courtesy of the Conservation Research Laboratory, Texas A&M University

Composite Artifact Example

Some of the objects conserved were composite artifacts. They were a combination of glass and pewter. In this example, we have fragments of the top of a green case bottle with a pewter screw-top lid (figure 7). After initial evaluation it was determined that the glass was the most fragile component of the piece and should be treated first. After an initial cleaning to remove excess sand, the artifact was desalinated followed by a series of alcohol baths and then placed in the polymer solution for final treatment. Once the object completed treatment, the pewter was assessed for any possible damage caused by the polymer solution. After visual inspection, it was determined that the pewter was undamaged by the silicone process and that it was possible to continue treating other glass and pewter composite pieces without risking the safety of the pewter.

Figure 7. Composite artifact, case bottle with pewter lid. *Photography courtesy of the Conservation Research Laboratory, Texas A&M University*

Final Comments

Results will vary by piece of glass. While there has been success in stabilizing multiple forms of glass at various levels of degradation, the final appearance of the glass varies from piece to piece. The appearance many times depends solely on the glass and its condition. Sometimes there is nothing more that can be done in regards to the appearance other than consolidation. When I say appearance, I mean the visible presence of layers of dead glass and iridescence. The piece may look fine once the process is in the last few stages, however once complete the natural appearance of the inner clouding and outer iridescence may become visible. These conditions are present in the glass before treatment begins, we are simply unable to visually observe them due to the optical illusion created by the liquids the glass is stored and treated within. The conservator does not visually know the actual condition of the glass until the treatment has been completed (Corvaia et al 1996).

When weighed against the IIC guidelines and the suggested guidelines for selecting an adhesive and/or consolidant for glass conservation, the silicone technique fulfills nearly every suggestion. Application of the silicone technique shows a respect for the integrity of the object in that there are circumstances where traditional methods are not adequate to successfully conserve an object as was the case with the hourglass. This research has provided an additional method that is successful with highly degraded and compromised vitreous materials. This not only shows an interest in the object, a respect for the integrity of the object, but it also fulfils the IIC guideline of continuing ones conservation education when the known methods are not enough. In the CRL and APRL, the conservators continue seeking alternatives to the traditional methods.

The silicone treatment has also proven to be a suitable treatment for conserving archaeological glass materials recovered from a marine site as based on suggested treatment selection guidelines (Newton and Davison 1989). The treatment is unobtrusive, adheres to glass, does not put any undue stress on the object, and adjusts to object movement. It is clear, does not yellow and remains stable for over 100 years. However, the silicone treatment is not an adhesive, it is only a consolidant. Therefore it does not “set” as is suggested in the selection guidelines. That said, the treatment does allow for the use of an adhesive. One unanticipated bonus with the silicone is that if the application of the adhesive is incorrect the silicone allows for the piece to be disjoined without damage to the edges of the glass.

One area that is frequently mentioned in the conservation field is reversibility. I will briefly touch on that concept here. In reference to Applebaum's (1987) article "Criteria for Treatment: Reversibility," it is noted:

> Reversibility is often used inaccurately as a catch-all term for a variety of treatment criteria. These include such varied issues as the appropriateness of a treatment material to the aesthetic requirements of the object and the compatibility of a treatment material to the physical requirements of the object.

Applebaum (1987) argues that the use of the term reversibility should be confined to a process rather than to a material. "The idea that a material can be reversible is not logical," because remnants of materials used for conservation are always left behind (Horie 1983). As Romich et al (1995) state "reversibility, a general requirement in conservation, is limited in practice for consolidants as it is rarely possible to remove any type of fixation material completely from the body of a friable object. Accepting the fact that at least some of the consolidant will remain in the object indefinitely." This includes the most commonly used glass conservation material today, paraloid B-72. The real question is not if a material can be reversed. All logical conservators understand you cannot return an object to a previous state once treatment begins. This includes the state prior to cleaning. Once something has been removed or altered in some manner, the object is forever changed. This is where I would encourage conservators to once again refer to the IIC guidelines. By treating the object with an otherwise non-traditional material or process, such as the silicone technique, does the conservator have the integrity of the object in mind? Prior to applying the silicone treatment, is it within the knowledge and capabilities of the facility and the conservator to apply such a treatment? Is the conservator treating the object with a "single standard" as he/she would treat all artifacts? Is the treatment suitable for the object? And does the conservator continue educating himself/herself on the most recent possibilities for treatment? If the conservator can answer yes to these questions prior to applying the silicone technique then I argue that he/she is following the appropriate ethical guidelines for utilizing the silicone technique for archaeological materials. Rather than asking about reversibility, we should be asking about retreatability. There are two examples from this research that say we can retreat artifacts conserved with the silicone technologies, both in the case of retreating the silicone technique itself (10% solution) and retreating an object (the hourglass) previously treated with another conservation material. Both retreatments were successful.

This is not an argument for or against the use of silicone technologies. There is still much to be learned from this technology and many questions we have yet to answer. However, at this time and from this research, I do support the use of silicone polymers as a conservation tool for some archaeological glass materials. The final question is what will you do when faced with the choice of losing an object now or having the opportunity to apply a nontraditional technique to prolong the life expectancy of the object? This is a question that must be weighed for each individual conservator, institution and for each artifact. Sometimes choosing to treat is the only viable option.

CHAPTER VIII

CONCLUSION

This research was completed in 1999. At that time, the silicone oil technique and its application to waterlogged archaeological material was in its infancy at Texas A&M University. Many conservators were cautiously hopeful and others were strongly resistant to a technique that "impregnated" material with silicone polymers, was "crosslinked," and was "irreversible." While these concerns are not without merit, the research has shown that the application of the silicone oil technique by a knowledgeable conservator to waterlogged materials prolongs the life expectancy and many times enhances the appearance of the object.

This project was the first large scale application of silicone polymers and silanes to 17th-century archaeological glass recovered from a marine site. It was also the first time the chemicals Q-1 3563 and Q-9 1315 were combined to conserve archaeological glass and was completed through a "willingness to explore treatment parameters and combinations of polymers" (Smith 2003) and with an interest in expanding conservation options. Through this research we were able to answer a number of questions about the use and application of the silicone technologies and we confirmed that these materials are a viable resource for glass consolidation and conservation. Prior to the development of the passivation polymers technique, our selection of conservation materials that were sufficient for the treatment of archaeological glass recovered from marine sites was limited. Today, we have another option.

Since the time of this research a third method of silicone technologies application for archaeological glass has been designed and applied by Dr. Helen DeWolf of the CRL. This method returns to the original chemicals that initiated this journey, SFD-1 and MTM, but calls for a different chemical concentration and does not require the use of a vacuum chamber. To date, we now have three separate silicone technology methods utilizing different chemical concentrations and applications for archaeological glass recovered from marine sites. If we are to truly challenge the viability of the silicone technologies as applied to archaeological glass then the investigation needs to move beyond our findings and out into independent laboratories for testing, questioning and application.

GLOSSARY

Alkali	A soluble salt, one of the essential ingredients in making glass, being 15-20% of the batch. It serves as a flux to reduce the fusion point of silica.
Annealing	The process of reintroducing the glass object into an auxiliary part of the glass furnace, in order to cool it slowly to prevent stress cracks.
Batch	The mixture of raw materials, generally silica, soda and lime heated together in a crucible or pot to make glass. broken glass(cullet) and minor ingredients such as colorants may be added.
Core-forming	The technique of forming a vessel by trailing molten glass over a core supported by a metal rod. The object was removed from the rod and annealed, after which the core was scraped out.
Cullet	Clean, broken glass from glass objects discarded during manufacture, melted together with the fresh ingredients of a new batch.
Crucible	A heat resistant container used chemical reactions and melting materials at a high temperature.
Devitrification	Development of crystallinity in glass with progressive loss of transparency due to the loss of alkali.
Diagenesis	Physical, chemical, or biological change undergone by sediment when it first deposits.
Efflorescence	The formation of a powder or crust on the surface of a Body.
Faience	A fired silica body containing very small amounts of clay and/or alkali. It is covered with a glaze, which may or may not occur interstitially to the silica grains of the body (frit).

Flux	A substance added to enamel colors so as to lower their fusion point during firing to below that of the glass body to which they are applied. Flux was also added to the batch in order to facilitate the fusing of the silica.
Frit	The granular product of the first stage of ancient glass or glaze making; When cool, the frit is ground and melted to produce a homogenous material.
Fulgurite	Glass formed on beaches from lightening striking silica sand.
Lamellae	A thin scale, plate or layer.
Natron(nitrum)	Sodium sesquicarbonate, obtained from the natron lakes north-west of Cairo, Egypt. It was used as the soda constituent in making Egyptian Glass.
Obsidian	A volcanic material which is the earliest form of natural glass used by humans.
Oxidation	The combination of oxygen with a substance or the removal of hydrogen from it. A reaction when an atom loses electrons.
Plastic Flow	The slow deformation of a materials shape due to the continued application of a force allowing it to change without fracturing.
Potash	Potassium carbonate, it’s an alternative to soda as a source of alkali in the manufacture of glass. Potash glass is slightly heavier than soda glass; it passes from the molten to the rigid state more quickly and is therefore more difficult to manipulate.
Silica	A mineral which is one of the essential ingredients of glass. The most common form of silica used in glass-making was (and still is) sand.
Soda	Sodium carbonate. It (or alternatively potash) is used as the alkali ingredient of glass. It serves as a flux to reduce the fusion point of the silica in making glass.

Soda Glass	Also known as soda-lime gas, this is one of the most ancient of glasses and one of the most common. Before the 18th century soda was added to the batch in the form of ashes obtained by burning certain marine plants, such as kelp and seaweed.Soda was and is still used as a flux throughout the Mediterranean. It is light in weight and appearance.
Stability	Chemical durability, resistance to weathering.
Weathering	Changes on the surface of the glass (caused by exposure to adverse conditions) which appear as dulling, frosting, iridescence, or decomposition.

Definitions provided in part by:
Jones and Sullivan, *The Parks Canada Glass Glossary*. National Historic Parks and Sites, Canadian Parks Service (1989)
Newton and Davison, *Conservation of Glass*. Butterworths (1989)

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APPENDIX A

LA BELLE GLASS ASSEMBLAGE TREATED WITH SILICONE OILS

Artifact #	Object	Number of Pieces	Color	Dimensions	Condition before treatment	Treatment	Results
1149	medium body fragments	2	brown	5.07cm x 2.4cm, 2.68cm x 2.67cm	laminated, milky surface, dead glass	Standard CRL dehydration, silicone treatment 10-15% cross	good
1234	body fragment	1	green	3.58cm x .79cm x .31cm	good, translucent, pitted	Standard CRL dehydration, silicone treatment 10-15% cross	good
1261	pocket mirror fragment	1	dark/black	4.89cm x 3.03cm	encrusted	Standard CRL dehydration, silicone treatment 10-15% cross	good
1294	pocket mirror fragment	1	dark/black	3.74cm x 1.19cm	typical mirror degradation pattern	Standard CRL dehydration, silicone treatment 10-15% cross	good
1300	pocket mirror fragment	1	dark/black	4.55cm x 3.19cm	NA	Standard CRL dehydration, silicone treatment 10-15% cross	good
1478	small fragment (possibly onion bottle base)	1	dark	2.65cm x 1.21cm x .39cm	good	Standard CRL dehydration, silicone treatment 10-15% cross	good
1484	small body fragment (possibly modern)	1	green	2.35cm x 1.21cm	good, slight curve	Standard CRL dehydration, silicone treatment 10-15% cross	good
1526	pocket mirror fragment (1/2)	1	dark/black	5.78cm x 3.12cm	good, slightly encrusted, small section/layer missing from back. crystal like material visible in section	Standard CRL dehydration, silicone treatment 10-15% cross	good
1590	fragments (5 large fragments, 6 small fragments	11	brown/dark	5.3cm x 2.3cm x .3cm, 3.9cm x 2.4cm x .2cm, 2.4cm, 2.6cm x .3cm, 5cm x 3.1cm, .2cm	fair, completely oxidized, opaque	Standard CRL dehydration, silicone treatment 10-15% cross	good
1721	bottle fragments	2	green/brown	3.38cm x 4.69cm, 3.16cm x 3.34cm	fair, oxidized on surface, surface pitting, side/corner of case bottle	Standard CRL dehydration, silicone treatment 10-15% cross	good
1833	cup handle	1	clear	3.74cm, 2.51 cm inside, 1.2cm & 1.21 cm points of attachment	good, surface pitting	Standard CRL dehydration, silicone treatment 10-15% cross	good
1848	case bottle fragment (shoulder/neck)	1	green	3.17cm x 3.96cm x .38cm	translucent, surface pitting	Standard CRL dehydration, silicone treatment 10-15% cross	good
1949	fragments	3	dark, clear, brown	3.87cm x 2.08cm, 2.25cm x 1.95cm, 1.83cm x 1.44cm, .98cm x 1.08cm	poor condition, severely devitrified, exfoliating	Standard CRL dehydration, silicone treatment 10-15% cross	good, glass in general poor condition overall prior to treatment
2101	fragment	1	brown	2.48cm x 1.32cm	exfoliating on outer layers, previous color green	Standard CRL dehydration, silicone treatment 10-15% cross	good
2102	body fragment	1	green	3.75cm x .35cm	good, translucent	Standard CRL dehydration, silicone treatment 10-15% cross	good
2108	body fragment	1	green/brown	2.59cm x 1.79cm x .13cm	fair, translucent, oxidation and iridescence visible on surface, pitting visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
2162	pocket mirror fragments, onion bottle fragments	3	dark	4.27cm x 3.71cm x .16cm, 2.66cm x 4.31cm x .49cm	fair, scratches visible on surface of mirrors, scratching and pitting visible on bottle surface	Standard CRL dehydration, silicone treatment 10-15% cross	good

2238	case bottle shoulder/side fragment	1	brown	8.18cm x4.79cm, .39cm	devitrified/fair	Standard CRL dehydration, silicone treatment 10-15% cross	good
2253	bottle body & rim fragments	11	dark	2.96cm x 2.13cm, 2", 1.55cm x 5.44cm, 6.44cm x 5.34cm x .33cm, 5.44cm x 3.12cm	rim w/ pewter ring devitrified, all other frags severely devitrified and delaminating	Standard CRL dehydration, silicone treatment 10-15% cross	good
2287.2	pocket mirror fragment	1	dark/black	2.8cm x 1.9cm	fair, one side covered in dead glass	Standard CRL dehydration, silicone treatment 10-15% cross	good
2290	rim fragment apothecary bottle (THC # 2505)	1	clear	2.96cm x 1.03cm x .18cm	good, rounded smooth edge	Standard CRL dehydration, silicone treatment 10-15% cross	good
2304	body fragment	1	green	3.84cm x 2.54cm	good/fair. Delaminated on edges, opaque, exterior surface exfoliating	Standard CRL dehydration, silicone treatment 10-15% cross	good
2333	small pocket mirror fragments	2	dark/black	3.16cm x 1.43cm, 2.22cm x 1.55cm	good, fit together	Standard CRL dehydration, silicone treatment 10-15% cross	good
2340	onion bottle body fragment	3	dark	2.6cm x 2.88cm, 3.1cm, 1.28cm,	fair	Standard CRL dehydration, silicone treatment 10-15% cross	good, shard broke into 3 pieces during conservation
2364	fragments (belong together)	2	dark	3.46cm x 2.9cm, 3.64cm x 2.96cm	poor. covered in dead glass	Standard CRL dehydration, silicone treatment 10-15% cross	good
2382	body fragment (THC #2208)	1	dark	1.32cm x 1.57cm	NA	Standard CRL dehydration, silicone treatment 10-15% cross	good
2396	body fragment	1	dark	2.79cm x 2.49cm	fair, dead glass visible within layers	Standard CRL dehydration, silicone treatment 10-15% cross	good
2418	onion bottle fragment, base/side	1	dark/green	8.62cm x 11.74cm x 1.2cm	fair, previously green, heavily encrusted, exfoliating in patches	Standard CRL dehydration, silicone treatment 10-15% cross	good
2497	case bottle body fragment (corner)	1	green	9cm x .3cm w (large side), 3.7cm (small side)	fair, delamination, oxidation, rust	Standard CRL dehydration, silicone treatment 10-15% cross	good
2497	case bottle fragment, side/corner (THC #2253)	1	green	3.9cm x 9.44cm x .49cm	good, translucent	Standard CRL dehydration, silicone treatment 10-15% cross	good
2529	body fragment	1	light green translucent	6.27cm x 5.32cm	good, concave, visible air bubbles, striations from manufacture	Standard CRL dehydration, silicone treatment 10-15% cross	good
2907	body fragments	6	dark		poor, dead glass fell off during conservation	Standard CRL dehydration, silicone treatment 10-15% cross	good
2935	pocket mirror fragment	1	dark	1.46cm x 3.45cm	typical mirror degradation pattern	Standard CRL dehydration, silicone treatment 10-15% cross	good
2938	concave fragments	5	clear		1pc lost during conservation, fragile, striations from glassmaking process visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
3055	pocket mirror fragment	1	dark	1.82cm x 1.05cm and .58cm		Standard CRL dehydration, silicone treatment 10-15% cross	good
3097	small body fragments	3	dark	3.4cm x 1.4cm x .5w, 1.9cm x 2.1 cm, 1.9cm x 1.4cm	very poor condition, oxidation, iron leaching	Standard CRL dehydration, silicone treatment 10-15% cross	good (16 pieces after conservation)

3104	pane glass	1	clear	4.56cm x 2.22cm	satisfactory, bubbles visible, surface rough	Standard CRL dehydration, silicone treatment 10-15% cross	good
3130	base of case bottle	1	dark	10.84cm x 10.99cm, 2.1 cm height of kick	large pontil scar visible, corner still intact, encrusted, went to Fort St. Louis	Standard CRL dehydration, silicone treatment 15% cross	good
3166	pocket mirror fragments	28	dark	lg 6.35cm diam, md 5.7cm	fair, frags=1 complete mirror, evidence of 7 other mirrors, 2 sizes	Standard CRL dehydration, silicone treatment 10-15% cross	good
3279	bottle rim fragments and curved body fragments	6	dark		fair	Standard CRL dehydration, silicone treatment 10-15% cross	good
3445	body fragment	1	brown	2.1cm x 2.2cm x .15thickness	fair	Standard CRL dehydration, silicone treatment 10-15% cross	good
3505	pocket mirror fragments	3	dark	2.36cm x 2.1cm & 1.5cm	fair, encrustation from knife blade	Standard CRL dehydration, silicone treatment 10-15% cross	good
3642	case bottle body fragments	2	dark	4.83cm & 4.13cm x 3.65cm	fair, lightly encrusted, surface degradation	Standard CRL dehydration, silicone treatment 10-15% cross	good
3663	body fragments	11	dark		fair, oxidized, lamellae,	Standard CRL dehydration, silicone treatment 10-15% cross	good
3668	body fragments	2	dark	2.29cm x 2.49cm and 1.54cm x 2.18cm	fair, layered w/oxidation throughout	Standard CRL dehydration, silicone treatment 10-15% cross	good
3673	onion bottle base/side fragment	1	dark	8.71cm x 4.09cm, 6.69cm	oxidized, lamellae visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
3681	body fragment	1	dark	2.69cm x 4.09cm	encrusted case bottle frag,THC#3255	Standard CRL dehydration, silicone treatment 10-15% cross	good
3690	body fragment	1	dark	2.97cm and 2.29cm x 1.90cm and 1.36cm	green w/oxidized glass	Standard CRL dehydration, silicone treatment 10-15% cross	good
3698	body fragment, corner/side fragment	2	dark/green	4.07cmx 2.09cm and 3.82cm x 1.68cm	corner/side translucent green w/dark areas	Standard CRL dehydration, silicone treatment 10-15% cross	good
3840	body fragment	1	dark	2.29cm x .52cm	good, layers visible at edge. Oxidation visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
3842	bottle base fragment	1	dark	2.96cm x 3.16cm	fair, lamellae visible, pitting, oxidation. Original color green	Standard CRL dehydration, silicone treatment 10-15% cross	good
3922	body fragment (possibly hourglass)	1	clear	3.82cm x 2.11cm	fragile, concave, striations from manufacture (THC 3276)	Standard CRL dehydration, silicone treatment 10-15% cross	good
3946	body fragment	1	dark	3.93cm x 3.27cm	milky spots w/oxidation	Standard CRL dehydration, silicone treatment 10-15% cross	good
4018	body fragments	2	lt green & dk green	2.46cm x 2.14cm and 1.57cm x 2.1cm	both translucent, lt green modern, dk green oxidized and laminated	Standard CRL dehydration, silicone treatment 10-15% cross	good
4165	small body fragments	3	black	1.5cm x 1.4cm, .9cm x 1.6cm	fair	Standard CRL dehydration, silicone treatment 10-15% cross	good
4192	pocket mirror fragments	2	dark	4cm x2.67 and 1.86cm	fair, 2 pieces belong together	Standard CRL dehydration, silicone treatment 10-15% cross	good

4299	body fragment	1	dark	3.94cm	poor, surface fragments detached during conservation, oxidized throughout. Edge/side, THC#3434	Standard CRL dehydration, silicone treatment 10-15% cross	good
4334	body fragment	1	brown	1.57cm x 4.75cm	slightly concave, layering at edges, pitting, manufacturing striations visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
4357	body fragments	2	dark	2.17cm x 1.52cm and 1.29cm x 1.05cm	oxidized throughout, THC #3453	Standard CRL dehydration, silicone treatment 10-15% cross	good
4412	pocket mirror fragments	4	dark		heavily encrusted, 2 pcs fit together	Standard CRL dehydration, silicone treatment 10-15% cross	good
4429	body fragment	1	dark	1.71cm x .94cm	oxidized, THC#2917	Standard CRL dehydration, silicone treatment 10-15% cross	good
4436	body fragment	1	clear	2.81cm x 2.24cm	clear concave w/patches of oxidized glass	Standard CRL dehydration, silicone treatment 10-15% cross	good
4501	body fragments	3	dark	4.48cm x 2.42cm and 2.43cm x 2.31 cm	2 pieces fit together, glass layered	Standard CRL dehydration, silicone treatment 10-15% cross	good
4512	body and base fragments	4	dark		1 onion bottle base frag, 3 devitrified body frags	Standard CRL dehydration, silicone treatment 10-15% cross	good
4543	body fragment	1	translucent green	2.72cm x 2.21cm	1 clear frag did not survive conservation	Standard CRL dehydration, silicone treatment 10-15% cross	good
4554	body fragment	1	amber translucent	4.38cm	concave modern piece, embossed	Standard CRL dehydration, silicone treatment 10-15% cross	good
4604	body fragments	2	dark	6.57cm x 2.94cm and 2.02cm x 1.75cm	lamellae visible, oxidized on surface	Standard CRL dehydration, silicone treatment 10-15% cross	good
4665	body fragments	2	dark	3.6cm x 4.79cm and 3.77cm x 1.77cm	previously translucent green, oxidized at time of conservation	Standard CRL dehydration, silicone treatment 10-15% cross	good
4670	body fragment	1	dark	1.17cm x .95cm	oxidized throughout	Standard CRL dehydration, silicone treatment 10-15% cross	good
4686	body fragment	1	dark	1.89cm x 2.25cm	slightly concave, some areas translucent. Oxidation present	Standard CRL dehydration, silicone treatment 10-15% cross	good
4762	case bottle edge/side	1	translucent green	3.32cm x 3.62cm	areas of dead glass, surface pitted & degraded	Standard CRL dehydration, silicone treatment 10-15% cross	good
4772	body fragment	1	translucent green	3.65cm x .99cm	good, striations from manufacturing visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
4795	body fragment	1	dark	1.82cm x 2.3cm	translucent prior to conservation, no longer translucent, oxidized throughout	Standard CRL dehydration, silicone treatment 10-15% cross	good
4849	case bottle body fragments	2	dark	2.85cm x 1.71cm & 2.41cm x 2.65cm	belong together, pitting and oxidation visible, THC #4150	Standard CRL dehydration, silicone treatment 10-15% cross	good
4891	pocket mirror fragments, body fragments	8	dark		some encrusted	Standard CRL dehydration, silicone treatment 10-15% cross	good

5108	base of hourglass	1	light green to clear	7.99cmx9.76cm, 2.52cm kick height	translucent, striations visible, possibly fits #5987	Standard CRL dehydration, silicone treatment 10-15% cross	good
5257	body fragments/rim and top of bottle	6	1 clear, 5 dark		clear concave frag, rim & top of bottle, body and corner pieces all oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good
5267	body fragments	5	dark		lamellae visible, oxidized on surface	Standard CRL dehydration, silicone treatment 10-15% cross	good
5275	body fragment	1	dark	1.61cm x 1.37cm	thin frag, original color green, pitting, oxidized on one edge	Standard CRL dehydration, silicone treatment 10-15% cross	good
5336	body fragments	2	dark	1.76cm x 1.55cm and 1.35cm x 1.65cm	oxidized throughout, fit together	Standard CRL dehydration, silicone treatment 10-15% cross	good
5363	body fragment	1	dark		during conservation small corner broke off, previously green, now oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good
5504.004	pocket mirror and pocket mirror fragments	19	dark	4.87cm x 4.99cm (intact mirror)	approx. 5 pocket mirrors	Standard CRL dehydration, silicone treatment 10-15% cross	good
5546	gin bottle fragments	13	dark green		base, top, fragments from middle, oxidation visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
5689	case bottle body fragment	1	green/brown	1.82cm x 1.73cm	edges devitrified, center translucent, THC#3682	Standard CRL dehydration, silicone treatment 10-15% cross	good
5839	body fragments	2	dark	1.78cm x .90cm and 2.79cm x 1.25cm	poor, thin, layered and oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good
5952	body fragment	1	translucent green	2.9cm x 3.11cm	good, concave manufacturing striations visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
5984	body fragment	1	clear	3.58cm x 2.78cm	concave, thin, manufacturing bubbles and scratches visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
5987	bottle or hourglass fragment	1	translucent light green	6.84cm	manufacturing striations visible, fits w/artifact #5108	Standard CRL dehydration, silicone treatment 10-15% cross	good
6133	pocket mirror fragment	1	dark	.62cm x 1.63cm	slightly encrusted	Standard CRL dehydration, silicone treatment 10-15% cross	good
6154	body fragment	1	translucent green	1.06cm x .74cm	some oxidation present on surface	Standard CRL dehydration, silicone treatment 10-15% cross	good
6188	body fragments	2	dark	1.79cm x 1.26cm and 1.08cm x .82cm	1 pc slight curve, both oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good
6217	fragments	6	green, clear, black	2.25cmx2.3cm, 1.5cm x 3.1cm, 1.5cm x 1.1 cm	fair to good	Standard CRL dehydration, silicone treatment 10-15% cross	good
6244	body fragment	1	clear	1.82cm x 1.35cm	good, concave, striations visible from manufacture	Standard CRL dehydration, silicone treatment 10-15% cross	good
6263	small case bottle in several fragment	33	green		fair, encrusted, slight pontil mark visible, oxidation visible on surface, cracks visible within matrix	Standard CRL dehydration, silicone treatment 15% cross	good, areas of glass prone to fracture
6324	body fragments	2	dark	.64cm x .86cm and 1.75cm x 1.64cm	1 piece slightly concave, both oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good

6364	bottom to small case bottle w/partial side	1	brown	base 7.57cm x 7.21cm. Kick 1.99cm	original color green, pontil scar visible, THC #3942	Standard CRL dehydration, silicone treatment 10-15% cross	good
6446	body fragment	1	translucent green	.58cm x 1.44cm	oxidized, rust color along edges	Standard CRL dehydration, silicone treatment 10-15% cross	good
6453	body fragments	4	dark		1pc is side of case bottle, original color green, lamellae visible on edges, THC #3773	Standard CRL dehydration, silicone treatment 10-15% cross	good
6463	body fragment, shoulder/neck frag	5	translucent green, brown		2 trans green, 3 brown, encrusted, oxidized	Standard CRL dehydration, silicone treatment 10-15% cross	good
6788	fragments	2	dark	.8cm x 1.27cm and .72cm x 1.5cm	nondiagnostic, poor, slightly concave, rust oxidation	Standard CRL dehydration, silicone treatment 10-15% cross	good
7255	fragment	1	dark	.91cm x 1.7cm	nondiagnostic	Standard CRL dehydration, silicone treatment 10-15% cross	good
7287	fragments of case bottle, neck shoulder, body	34	green/brown		Poor to moderate	Standard CRL dehydration, silicone treatment 10-15% cross	good
7288	kick of bottle	1		4.63cm x 4.78cm		Standard CRL dehydration, silicone treatment 10-15% cross	good
7377	small case bottle body fragment	1	brown	2.4cm x 1.5cm x .2cm thick	Poor to moderate	Standard CRL dehydration, silicone treatment 10-15% cross	good
7380	body fragment	1	dark	3.56cm x 6.36cm	poor, oxidized, devitrified, lamellae visible, previously translucent green	Standard CRL dehydration, silicone treatment 10-15% cross	good
7928	base fragment	1	translucent green	3.64cm	visible oxidation and layering	Standard CRL dehydration, silicone treatment 10-15% cross	good
7930	fragments	7	dark		poor, nondiagnostic, prev trans green	Standard CRL dehydration, silicone treatment 10-15% cross	good
10221	case bottle fragments w/base	13	dark		encrusted, oxidized glass throughout surface, large pontil scar visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
10258	case bottle w/pewter screw top lid	46	blue/green and yellow/green		composite artifact	Standard CRL dehydration, silicone treatment 10-15% cross	good
10258.2	hourglass fragment	1	clear	1.84cm x 4.0cm	concave, layering visible, slight oxidation visible	Standard CRL dehydration, silicone treatment 10-15% cross	good
10375	fragment (broke into several pieces)	11	dark		poor, oxidized, broke during conservation	Standard CRL dehydration, silicone treatment 10-15% cross	good
10799	pewter screw top lid w/neck glass and wooden stopper	1	green		good, composite artifact	Standard CRL dehydration, silicone treatment 10-15% cross, then placed entire object in ER for 10 days, Wooden stopper treated separately	good, glass protected in ER by previous silicone treatment, but now fragile
11126	case bottle body fragments	2	brown	6.1cm x 4.5cm, 7.1cm x 3.9cm	very poor condition, flaking, complete oxidation	Standard CRL dehydration, silicone treatment 10-15% cross	good
11642	body fragment	1	dark	2.44cm x 1.88cm	concave, oxidized, prev trans green.	Standard CRL dehydration, silicone treatment 10-15% cross	good

12533	Top of large case bottle w/pewter ring, small frags	5	Green	9.04cm x 11.06cm .23 to .14 thickness	Poor to moderate, composite artifact	Standard CRL dehydration, silicone treatment 10-15% cross	good
13012	case bottle fragments (3 fit together body, base/body)	10				Standard CRL dehydration, silicone treatment 15% cross	good, duplicate use of #
no tag	large case bottle w/pewter lid				visible oxidation on surface and crazing, composite artifact	Standard CRL dehydration, silicone treatment 10-15% cross	good, no apparent tag, # of fragments, missing data
NP	small body fragment	1	green	1.95cm x 2.15cm	moderate, pitting, areas of brown oxidation	Standard CRL dehydration, silicone treatment 10-15% cross	good
	Total number of artifacts conserved with the standard silicone and crosslinker solution pieces:	**436**					
7200	top of case bottle w/pewter neck	1	brown	11.85cm x 8.6cm	poor, glass soft to the touch, encrusted, oxidized, surface delaminated, composite artifact	Standard CRL dehydration, Silicone treatment 10% crosslinker	good
7722	body fragment	1	dark	1.05cm x 1.3cm	fair, rust oxidation, delaminated	Standard CRL dehydration, Silicone treatment 10% crosslinker	good
10752	pewter screw top lid w/glass	1	orange/brown		poor, oxidized and corroded, composite artifact	Standard CRL dehydration, Silicone treatment 10% crosslinker	good
11696	case bottle w/ screw top lid, collar, pane glass	47	green, clear	pane (4.32cm x 3.18cm)	pane good, manufacturing bubbles scratches visible, bottle fair, surface devitrified	Standard CRL dehydration, Silicone treatment 10% crosslinker	good
	Total # of successfully conserved pieces with a 10% solution	**50**					
	Retreated Artifacts						
5592	shoulder to case bottle	1	dark	4.1cm x 6.8cm	poor, oxidized, originally green	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
6183	body case bottle fragment	1	translucent green	2.4cm x 3.6cm	good, abrasions and scratching, lamination on edges, rust oxidation visible	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
6512	case bottle fragments, base, side, neck	3	dark		poor to fair, black oxidation present	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
6795	fragment	1	clear	.9cm & .5cm x 1.3cm	good, concave	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated	fair, retreat good

						w/15% Si dipping	
7793	fragments	2	green and dark	5.7cm x 2.8cm & 3.6cm and 4cm x 2.7cm & .2cm	green is good to fair w/delamination, slight curve one edge, dark is fair to poor, opaque w/rust oxidation present	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
10625	shoulder to case bottle	1	dark	7.6cm x 3.45cm	fair to poor, black oxidation, visible devitrification	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
11160	body fragments	10	dark		poor, completely oxidized, 8pcs fit together	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
11472	onion bottle base fragment	1	dark	5.1cm x 4.9cm	fair, iron leaching, concreted, corrosion outerl layer	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
11595	possibly base of bottle kick	1	dark	3.25cm x 3cm	fair, oxidized, concreted slightly concave	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good
13012	case bottle fragments, side, side/corner, base/corner	3	dark		fair to poor, edges friable, encrusted, rust present, surface oxidized	Standard CRL dehydration, Silicone treatment 10% crosslinker. Retreated w/15% Si dipping	fair, retreat good, duplicate use of #
	Total # of retreated pieces	**24**					
2043	Onion bottle	1	dark		good, heavily encrusted, some devitrification	Standard CRL dehydration, Si vac 5 hrs, day 2 vac 8 hrs, catalyst in oven, catalyst & si dip (15% cross)	good

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